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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :

JUERGEN SCHUBERT, ET AL.

: EXAMINER: CAIN, E. J.

SERIALNO: 09/934,471 :

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: GROUP ART UNIT: 1714

FOR: PROCESS FOR COVERING  
SILICAS WITH WAX :DECLARATION UNDER 37 C.F.R. § 1.132COMMISSIONER FOR PATENTS  
ALEXANDRIA, VIRGINIA 22313

SIR:

Now comes Dr. Jürgen Schubert who deposes and states:

1. That I am a graduate of Chemistry and received my doctoral degree in the year 1992.
2. That I have been employed by Degussa for 11 years as a chemist in the field of performance silica and matting agents.

Now comes Reinhard Behl who deposes and states:

1. That I am a graduate of Realschule and received a Mittlere Reife degree in the year 1972.
2. That I have been employed by Degussa for 32 years as an engineer for paints and coatings in the field of paints and coatings.
3. That the following experiments were carried out by us or under our direct supervision and control.

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A silica according to the present invention was prepared as set forth below and had the properties as set forth below.

A precipitated silicic acid was intimately mixed with a PE wax [AF 30 (BASF)] in the ratio of 6 parts of wax and 94 parts of silicic acid. This mixture was proportioned into an impact air separation unit (ZPS 50). The impact air separation mill was operated with preheated grinding air during grinding. The inlet and exit temperature of the grinding air from the mill is set forth below in Table 1. The grinding settings (speed of air separation wheel and mill) are not critical for coating the silicic acid with wax, but are chosen such that the product has a distribution suitable for the desired flatting efficiency.

Characteristics of silicic acid used:

BET surface (m <sup>2</sup> /g)	450	
Mean particle size (μm)	27	(measuring instrument: Coulter LS 230)
Tamped density (g/l)	180	(not sieved)
Drying Loss (%)	6.0	(2 h at 105 °C)
Ignition Loss (%)	5.0	(2 h at 1000 °C)
pH	6.0	(5% in water)
DBP absorption (g/100 g)	335.0	

Table 1

Inlet temperature	(°C)	140-160
Outlet temperature	(°C)	120
Wax name		AF 30
d10*	(μm)	4.48
d50*	(μm)	6.49
d90*	(μm)	13.96
C content	(%)	5.65
Grinding value	(μm)	25

Gloss at 60° relative to standard**	(%)	-5.5
Wax detachment		None

\* Measuring instrument: Coulter LS 230

\*\* Standard: Acematt HK 460, Degussa AG

A sedimentation test was carried out as follows:

4.76% by weight of the silica described above was dispersed in 95.24% of an alkyd urea lacquer equivalent to the alkyd urea lacquer described on page 3 of EP 0541359 A1 for 10 min. in a 350ml PE-beaker using a wing agitator with a diameter of 45mm at 2000 rpm. The mixture was transferred into a laboratory bottle and sealed. The bottle was subsequently placed and stored in a water bath at 70°C. The sedimentation behavior was observed after 0, 4, 7, 12 and 19 days. The results are summarized in Table 2:

Table 2

storing time [days]	Phase separations observed?	Height of the silica phase of the overall height	Hard sediment observed after 19 days?	Soft sediment observed after 19 days?	Gelling observed after 19 days?
0	no	100			
4	yes	68			
7	yes	65			
12	yes	58			
19	yes	57	no	yes	no

#### Discussion of the Results of the Experiments.

The results get forth under "Phase separations observed?" and "height of the silica phase of the overall height" are discussed below and the meaning of the headings will be explained with the assistance of the following figures.

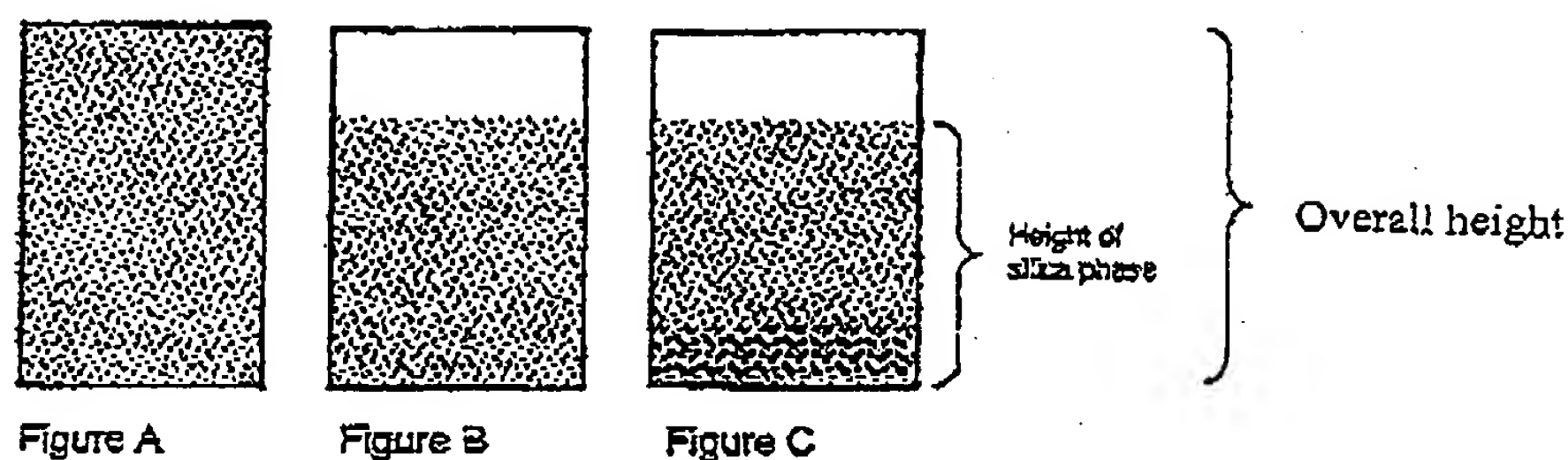


Figure A: Represents a lacquer comprising freshly dispersed silica

Figure B: Represents the lacquer of Figure A after some days storing. It is apparent that a phase separation has occurred but the silica is still completely dispersed. No sediment is observed.

Figure C: Represents the lacquer of Figure A and B after further storing. A sediment has now formed.

The values given in Table 2 under the heading "Height of the silica phase of the overall height" give an impression of how far the phase separation has progressed after 0, 4, 7, 12 and 19 days.

The data in Table 2 demonstrate that the silica prepared according to the process of the present invention possesses improved properties, as compared to the silica prepared and tested in Table II of EP 0541359 A1 on pages 7-9 of the reference with the inlet air temperature at 180°C as specified on page 6, lines 41-43 of EP 0541359 A1. The silica of test Example I of Table II of EP 0541359 A1 shows a soft sediment up to 7 days with hard sediment assumed to start at 8 days. The silica of the present invention shows a soft sediment at 19 days and no hard sediment at 19 days. Test Examples 2-7 of Table II of EP 0541359 A1 show hard sediment forming in a period of 24 hours to 6 days in contrast to the formation of no hard sediment at 19 days with the silica of the present invention.

Further, no gelling was observed with the silica prepared according to the process of the present invention. All sample mixtures of the silica of the present invention could be stirred easily, even after 19 days. In contrast, Examples 1-3, 6 and 7 of EP 0541359 A1 showed thickening and/or gelling in a period of time from 6 hours to 3 days.

The following tests clearly demonstrate the superiority of the silica prepared according to the process of the present invention in resistance to sedimentation and gelling, as compared to silica prepared by the process according to EP 0541359 A1.

4. The undersigned petitioners declare further that all statements made herein of their own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing therefrom.

5. Further deponent saith not.

  
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